Esterification of Free Fatty Acids in simulated Cooking Oil Using Sulphuric acids as catalyst: - Pre-treatment of Feedstock for Biodiesel Production

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ABSTRACT

The objective of this research is to study the batch kinetic of FFAs effect of esterification by evaluating the parameters over a wide range operation conditions those are, effect of catalyst loading and methanol to oil feed molar ratio and time effect. The scope of the research is focus on limited to raw material of the simulated cooking oil by changing the parameters such as catalyst loading, methanol to oil molar ratio and time effect. Study will be focusing only for pre-treatment process. In this paper raw material (virgin oil) is to be simulated with free fatty acids to study the batch kinetic of FFAs effect of esterification by evaluating the parameters over a wide range operating conditions those are elimination of mass transfer resistance, effect of catalyst loading, methanol to oil feed molar ratio and time effect. The Gas Chromatography (GC) equipped with HP-5 silica capillary column will be used in this research also with the titration technique to get the FFA reading. The research uses sulphuric acid as the catalyst. With proper research focus and development, waste cooking oil can indeed become the next ideal feedstock for biodiesel.

ABSTRAK

Objektif kajian ini adalah untuk mengkaji kitetik batch kesan FFA kepada proses esterifikasi dengan menilai parameter-parameter pada beberapa keadaan iaitu kesan peratusan katalis, nisbah methanol kepada minyak, dan kesan terhadap perubahan masa.Skop kajian ini member focus kepada bahan mentah iaitu minyak kelapa sawit yang terpakai yang diubahkan dengan menukar parameter- parameter tersebut.Kajian hanya member fokus terhadap proses awal rawatan iaitu esterifikasi. Dalam kajian ini minyak masak akan dicampur asid oleik untuk menambah kandungan FFA untuk mengkaji kinetik batch kesan FFA terhadap parameter-parameter tersebut. Gas kromatografi yang dilengkapi kolum kapilari silica HP-5 akan digunakan dalam kajian ini bersama dengan pentitratan teknik untuk mendapat bacaan FFA. Asid Sulfurik akan digunakan dalam kajian ini sebagai katalis. Dengan kajian yang baik dan focus minyak masak terpakai boleh menjadi bahan mentah seterusnya yang ideal dalam penghasilan biodiesel.

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LIST OF ABBREVIATIONS

FFA	Free fatty acid
FAME	Fatty acid methyl ester
WCO	Waste cooking oil
wt.	Weight

1 INTRODUCTION

1.1 Motivation and statement of problem

Biodiesel can be used by can be used by blending with petroleum biodiesel or can be directly used itself. Biodiesel, is generally produced by the processs of transesterification. Transesterification which is to derive alkyl esters from triglycerides (TGs). There is the problem with the transesterification alone as the process which the conventional biodiesel production technology involves the use of alkaline homogeneous catalysts such as NaOH and KOH.They are not compatible for feedstocks with large amounts of free fatty acids (FFAs) and moisture. It will forms soap and affecting the feasibility of glycerol separation. There is an alternative solution that is allow the feedstocks with a large amount of FFAs such waste cooking oil then allow for low cost biodiesel production. But it need to be done by two steps of process in order to make it fast as 1000 times rather than the single step. The first step is called the pretreatment which is esterification of FFAs then the transesterification of TGs to produce biodiesel. The esterification process however conventionally carried out by using some hazardous and polluting corrosive acids such as HCl and H_2SO_4 and they need to be replaced with the safer solid acid catalysts. Heteropoly acids (HPAs) seems to be the solution, by offering more efficient and cleaner processes. Needed to be considered is the stability whether in thermal situation and polar media, surface area, type of reactions it catalyze, reaction temperature and also needed to know their disadvantages so it can be overcome.

1.2 Objectives

This research is aimed to study the batch kinetic of FFAs effect of esterification by evaluating the parameters over a wide range operating conditions those are effect of time on reaction, effect of catalyst loading, and methanol to oil feed molar ratio.

1.3 Scope of this research

The research scopes scopes for this study focus limited to raw material of the simulated cooking oil by changing the parameters such as effect of time on reaction, catalyst

loading, and oil to methanol molar ratio. Study will be focusing only for pre-treatment process.

1.4 Main contribution of this work

The main contribution of this work will be to analyze the effectiveness and the optimum conditions for esterification of free fatty acids in simulated waste cooking oil using sulphuric acids as catalyst. Furthermore, to lesser dependant on fossil fuel, to avoid competition between plants for food and for biodiesel and to utilize used cooking oil rather being disposed.

1.5 Organisation of this thesis

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides a brief introduction and literature review regarding the research project from global perspective to economic standpoint and scientific viewpoint. This literature review also provides a discussion of the abundant of suitable biodiesel feedstock, transesterification reaction, esterification reaction and some of catalyst used for biodiesel production. A summary of the previous experimental work transesterification reaction, esterification reaction and catalyst used for biodiesel production is also presented.

Chapter 3 gives a review of the esterification reaction, setup process and how the experiment is run in complete step by step procedures. The method to evaluate the performances of the reaction with three different parameters in similar condition by varying the value of each parameter to obtain the experimental data.

Chapter 4 is devoted to data that gathered from the experiments done and discussion regarding the results that are obtained. A brief review of the plotted graphs from the experimental values from each parameter. A detailed discussion concerning the conditions for esterification of free fatty acids in simulated waste cooking oil using sulphuric acids as catalyst.

2 LITERATURE REVIEW

2.1 Overview

This paper presents the experimental studies for esterification of free fatty acids in simulated waste cooking oil using sulphuric acids as catalyst. A brief introduction and literature review regarding the research project from global perspective to economic standpoint and scientific viewpoint. This literature review also provides a discussion of the abundant of suitable biodiesel feedstock, transesterification reaction, esterification reaction and numerous of catalyst used for biodiesel production. A summary of the previous experimental work transesterification reaction, esterification reaction and catalyst used for biodiesel production is also presented.

2.2 Introduction

Biodiesel is as an alternative fuel for diesel engines, it is biodegradable, nontoxic and renewable. Renewable energy has been highlighted in the last ten years due to its potential to replace fossil fuel especially for transportation. Renewable energy sources such as solar, wind, hydro, and energy from biomass and waste has been successfully developed and used by different nations to limit the use of fossil fuels. Nevertheless, based on recent study from International Energy Agency (IEA), only energy produced from renewable sources and waste has the highest potential among other renewable resources as shown in Fig. 1. (International Energy Agency I, 2008)

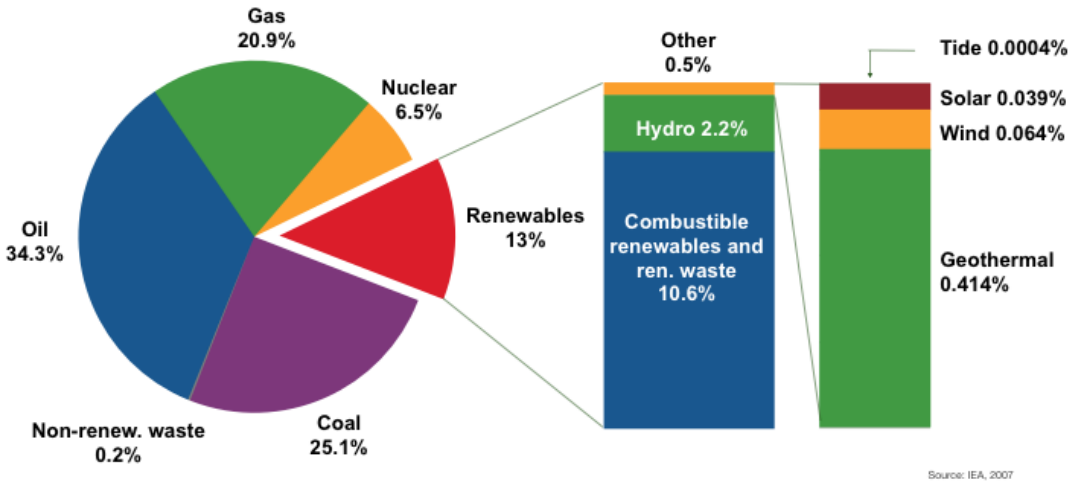

Figure 2.2a: World total energy supply by fuel

To date, fossil fuels account over 80.3% of the primary energy consumed in the world, and 57.7% of the amount is used in transportation sector (International Energy Agency I, 2006). On the other hand, the global consumption of diesel fuel is estimated to be 934 million tonnes per year (Kulkarni and Dalai, 2006). Thus, the World Energy Forum predicted that fossil oil will be exhausted in less than 10 decades, if new oil wells are not found (Sharma and Singh, 2009). The main reason that caused the fast diminishing of energy resources is due to rapid population and industrialization growth globally (Pimentel and Pimentel, 2006). Due to this phenomenon, the era of cheap crude oil no longer exist leading to high sky rocketing price of petroleum, bellicose conflict and increasing the number of undernourished people especially from undeveloped countries. Fig.1.2 presents the projection of world energy demand in the near future indicating that there is an urgent need to find more new renewable energies to assure energy security worldwide (Exxon Mobile, 2004).

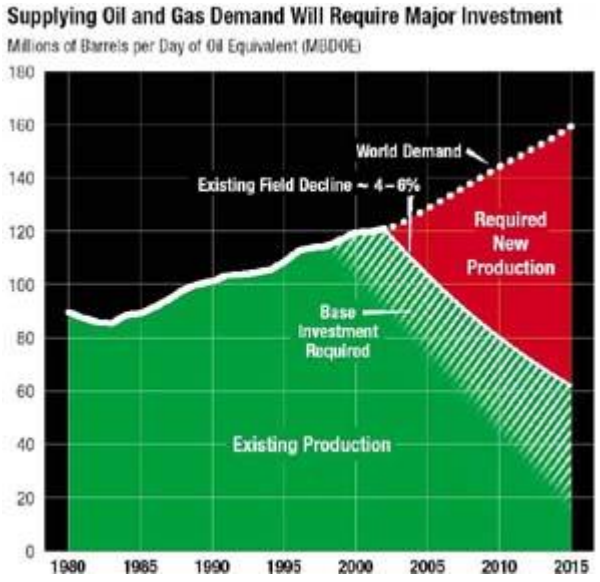

Fig. 2.2b: Projection of energy demand for the near future

2.3 Energy

Energy is an indirectly observed quantity that is often understood as the ability of a physical system to do work on other physical systems. It is something that human very concerning about, and everyday people consume energy. It is important that energy gives so much importance in human life to make a convenience life. The types of energy are kinetic, sound, thermal(heat), chemical, electrical, and gravitational energy.

There are also some renewable and non-renewable energy. Energy exists freely in nature. Some of them exist infinitely (never run out, called renewable), the rest have finite amounts (they took millions of years to form, and will run out one day, called non-renewable**).** Non-renewable energy is energy from fossil fuels (coal, crude oil, natural gas) and uranium. Notice that water, wind, sun and biomass (vegetation) are all available naturally and were not formed. The others do not exist by themselves, they were formed. Renewable energy resources are always available to be tapped, and will not run out. This is why some people call it green energy.

2.4 Diesel

Diesel fuel in general is any liquid fuel used in diesel engines. The most common is a specific fractional distillate of petroleum fuel oil. Petroleum-derived diesel is increasingly called petrodiesel. Diesel is usually used as a vehicles fuel and car also. It is a type of energy which can moves vehicle which is a kinetic energy. Diesel-powered cars generally have a better fuel economy than equivalent gasoline engines and produce less greenhouse gas emission. Their greater economy is due to the higher energy per-litre content of diesel fuel and the intrinsic efficiency of the diesel engine. While petrodiesel's higher density results in higher greenhouse gas emissions per litre compared to gasoline, the 20–40% better fuel economy achieved by modern diesel-engined automobiles offsets the higher per-litre emissions of greenhouse gases, and a diesel-powered vehicle emits 10-20 percent less greenhouse gas than comparable gasoline vehicles. Biodiesel-powered diesel engines offer substantially improved emission reductions compared to petrodiesel or gasoline-powered engines, while retaining most of the fuel economy advantages over conventional gasoline-powered automobiles. However, this fuel has the limitation due to the reserve of world’s oil will not remain forever. So, something new fuel must be produced as the alternatives of diesel and it is renewable energy. Biodiesel is a promising as the alternatives of diesel.

2.5 Biodiesel

Biodiesel is an alternative fuel for diesel engines. Biodiesel refers to a vegetable oil- or animal fat-based diesel fuel consisting of long-chain alkyl (methyl, propyl or ethyl) esters. Biodiesel is typically made by chemically reacting lipids (e.g., vegetable oil, animal fat (tallow) with an alcohol producing fatty acid esters .Biodiesel is meant to be

used in standard diesel engines and is thus distinct from the vegetable and waste oils used to fuel converted diesel engines. Biodiesel can be used alone, or blended with petrodiesel. Biodiesel can also be used as a low carbon alternative to heating oil.

Pure biodiesel is non-toxic, biodegradable and greenhouse gas neutral. As a blended addition to Ultra Low Sulphur Diesel, Biodiesel reduces pollution, particulates (soot) and hydrocarbons and restores lubricity lost due to the reduction of sulphur compounds in ULSD (thereby reducing engine wear). Both Biodiesel and mineral diesel have similar energy and power content and have proven to be of a similar retail value at the pump.

Biodiesel has emerged as a realistic and desirable alternative, or blended addition, to mineral diesel and is becoming an increasingly valuable contributor to the world's drive to reduce greenhouse gas emissions. It has been in general pure use for the last 10 years in continental Europe. However, the majority of UK Biodiesel production is expected to be used as a 5-20% blend with mineral diesel. In the UK, a 5 per cent. Biodiesel blend is currently being marketed on forecourts; Petroplus, a significant diesel refiner, markets "Bioplus" and Greenergy, a significant distributor of green fuels (25 per cent. owned by Tesco plc), markets "Global Diesel". Biodiesel, when added to ULSD, restores lubricity lost due to the reduction of sulphur compounds, as well as adding environmental benefits to the blend. Pure Biodiesel has many other advantages including, non toxic and biodegradable; it is fully degraded in a waterway environment within approximately 28 days.

Significant lubricant characteristics enabling a reduction in wear and extended efficiency for injectors and for all engines using ULSD, resulting in lower maintenance costs.

Biodiesel also come with few disadvantages. Biodiesel gels in cold weather. Biodiesel, just like petroleum diesel does gel in cold weather but the exact temperature that it will gel at will depend on what it was made from. The fuel will gel at around the same temperature than the oil or fat that was used to make it. So if for instance you used tallow or animal fat that is solid at room temperature to make your fuel from chances are good that your biodiesel will also be solid at room temperature. Biodiesel from Canola oil on the other hand will only start gelling at 14°F (-10°C) which is pretty much the same as conventional petroleum diesel.

Biodiesel is surrounded by negative sentiment, confusion and fear. This the most serious of all the disadvantages of biodiesel and the one that is the hardest to find a

solution to. The Negative sentiment and fear that surrounds it, fear that if use biodiesel in a car will brake it and void the warranty.

Food versus Fuel. Is it right when we use something that could have fed a hungry child to power trucks? This is a big concern and something that people constantly talk about, do we plant and grow food or fuel.

2.6 Feedstocks

Biodiesel, it can be refers to a vegetable oil or animal fat. The animal fat although they make great fuel with some cold weather issues the problem with animal products is the smell. They are best avoided. One of the other issues with rendered animal fats are that they tend to have a high free fatty acid content. Usually this is caused by the fats breaking down during storage or rendering which uses high temperatures. Chicken fat tends to make pale yellow biodiesel with relatively good cold weather properties while mammal fats tend to make biodiesel with poor cold weather properties.

Also corn can be the feedstock, corn is a starch crop but does have a small amount of oil in it. Corn oil does make good biodiesel but it is not a good crop to grow specifically to make biodiesel from. If the crop is fermented and turned into Ethanol then the oil can also be separated and used for making Biodiesel in which case it may be viable.

The algae as raw material, A number of species of Algae have up to 60% oil content. This together with the very high yields per Hectare for Algae makes it a very exciting possible crop for biodiesel production. Algae to biodiesel is still very much in the research and development stage.

Kumaran et al. (2011) highlighted that edible oil as feedstock which constitutes 80%-95% of the overall production cost is depend on its supply and reliable quality. However, Kulkarni and Dalai (2006) pointed that commercialization of biodiesel production from vegetable oil has big obstacle due to high manufacturing cost as virgin vegetable oil as raw material may cause a fortune and the production cost is proportional to the feed, plant size, and value of the glycerine byproduct. Furthermore, Gui et al. (2008) precisely bring up that there are many claims that a lot of problems may arise since more than 95% of the biodiesel is made from edible oil. Thus, large-scale of food resources are actually being converted into automotive fuels creating imbalance to the food supply and demand market.

In the Mediterranean countries, there is an abundant production of olive pomace oil which is a non edible oil obtained from the olive pomace mainly originating from Peloponnesus and Crete regions (Che et al., 2012). In addition, Olive pomace, which can be use as low cost support is an industrial byproduct of the olive oil production process, is obtained by squeezing the olive pulp usually further extracted with *n*-hexane yielding olive pomace oil (Yücel, 2011). Methods to produce biodiesel using the olive pomace as feedstock involving immobilized enzymes were also reported and seemed to be an available process despite a relatively complex work up (\ Alrouh et al., 2012). However, the high cost of enzyme remains a barrier for its industrial applications (Yücel, 2011) and a recent study on the concentrations of polycyclic aromatic hydrocarbons (PAH) in olive pomace oil revealed that the oil is hazardous to the human health due to the high PAH content (Che et al., 2012). In India, *Jatropha curcas* (jatophra) and *Pongamia pinnata* (karanja) are widely use to produce biodiesel with seeds possess 40% and 33% oil in each and both contain toxins and hence are non-edible (Sharma and Singh., 2008).

Biodiesel produced from waste cooking oil has the potential to reduce the green house gases (GHG) emission for up to 86% compared to soy based biodiesel which only results in 57% reduction of GHG compared to fossil fuel (Kumaran et al., 2011). The waste cooking oil is obtained after several times of frying will contribute to the change in chemical and physical properties of the oil. Therefore the composition will be slightly different compared to the virgin oils. Boffito et al. (2012) claimed that the high waste cooking oil potential is also recognized by the EU (European Union) directive 2009/28/EC, from common vegetable oils, the greenhouse emission savings range from 36% to 62% based on the type of oils source, where waste vegetable or animal oil is reported to save about the 88% of greenhouse emissions, a quite high value if compared to biodiesel from virgin oil.

2.7 Process of biodiesel production

To reduce the viscosity of of vegetable oils, methods have been developed such as dilution (blending), micro-emulsification, pyrolysis (thermal cracking), and transesterification. Pyrolysis is a method of conversion of one substance into another through heating or heating with the aid of the catalyst in the absence of air or oxygen. It

involves heating in the absence of air or oxygen and cleavage of chemical bonds to yield small molecules. The material used for pyrolysis can be vegetable oils, animal fats, natural fatty acids and methyl esters of fatty acids. The liquid fuel produced from this process has almost identical chemical components to conventional diesel fuel.). Micro-emulsification is another approach to reduce the viscosity of vegetable oils. It is a thermodynamically stable dispersion of oil in water and water in oil. Both used surfactant as the principal agent to enable water and oil to mix. Crude vegetable oils can be blended directly or diluted with the diesel fuel to improve viscosity. Dilution reduces the viscosity, engine performance problems such as injector coking, and more carbon deposits. However, dilution is not suitable for long term use in a direct injection engine. Pure biodiesel or 100% biodiesel is referred to as B100. A biodiesel blend is pure biodiesel blended with petrodiesel. Biodiesel blends are referred to as BXX. The XX indicates the amount of biodiesel in the blend (i.e., a B90 blend is 90% biodiesel and 10% petrodiesel).

2.8 Transesterification

Transesterification also called alcoholysis, is a chemical reaction of an oil or fat with an alcohol in the presence of a catalyst to form esters and glycerol. It involves a sequence of three consecutive reversible reactions where triglycerides (TG) are converted to diglycerides (DG) and then DG are converted to monoglycerides (MG) followed by the conversion of MG to glycerol. In each step an ester is produced and thus three ester molecules are produced from one molecule of TG.

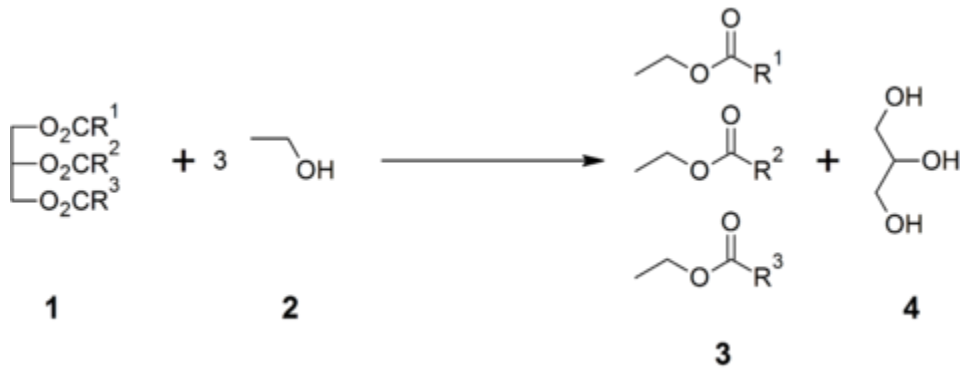

Fig.2.8:Triglycerides (**1**) are reacted with an alcohol such as ethanol (**2**) to give ethyl esters of fatty acids (**3**) and glycerol (**4**):

The conventional production by transesterification alone involves the use of alkaline homogenous catalysts such as NaOH however not compatible for feedstocks with large amounts of free fatty acids (FFAs) and moisture due to the formation of soaps that strongly affect the feasibility of glycerol separation which is an important coproduct of the reaction. Liquid acids such as HCl and H_2SO_4 in the other hand need a very long time and a very high molar ratio of methanol to oil although it is found to be more efficient. Also it got problem with the corrosion of reaction vessels and problem of recycling.

2.9 Esterification

There is alternative finding for low cost of biodiesel production and it is interesting alternative. Waste cooking oil or used vegetable oil, it is the biodiesel raw material of choice. The cost is good, in many countries it is free and it is available with little work. Raw materials with low quality as feedstocks such as waste cooking oil which easily got which are rich with free fatty acids. Vegetable oils used in industrial or household frying undergo degradation by hydrolytic and oxidative raeactions, both processes being responsible for changes in the chemical and physical properties, as compared to the fresh oil (Costa Neto et al., 2000). During frying, the viscosity and acidity of the oil increases, and the oil becomes darker and develops an unpleasant odour. So, this will lead to two step process where the needed of the esterification of the high free fatty acid contained in the feedstocks.

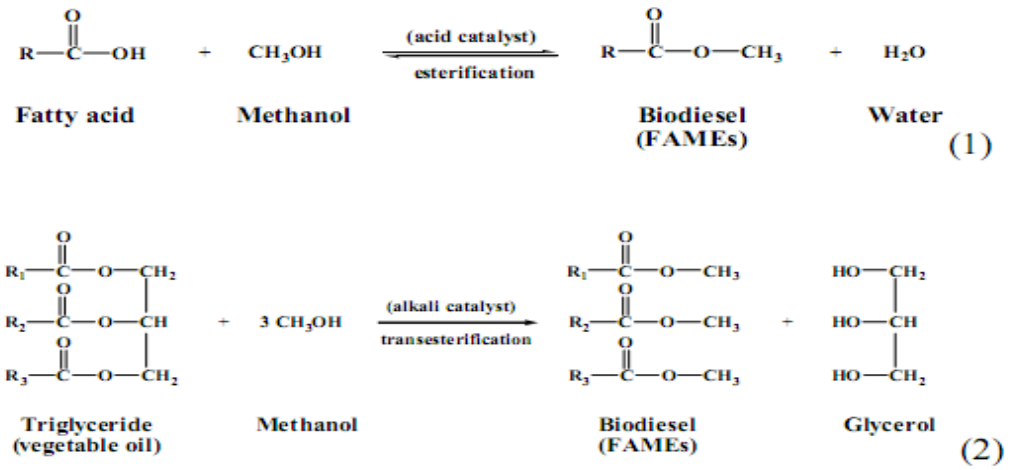

Fig.2.9: The 2 stages of production of biodiesel

The esterification as the first or pretreatment process. It is usually to carry out the esterification using various conventional mineral acid such as H_2SO_4, HF, H_3PO_4 and HCL also cannot use the alkali catalysts due to the formation of soap as waste cooking oil contain high FFA. The replacement of these hazardous and polluting corrosive liquid acids by solid acid catalysts is preferred and one of the major demands of society. Abiney L. Cardoso et al. (2008) confirmed ester hydrolysis can occur under either esterification or transesterification, esterification is usually believed to be more tolerant of water content and high free fatty acids levels in the initial feedstock and, thus, more suitable for low-grade fats and greases. Suyin Gan et al. (2010) proposed the esterification rate was fast when the water content was minimal. This is because, as the forward reaction continued, water content in the reaction mixture gradually increased due to high production of water as a byproduct.

2.10 Catalyst

A catalyst substance or material that will change the rate of reaction without it being consumed by the reaction. In esterification process Although there are many types of catalyst such homogeneous catalyst and heterogeneous catalyst. They can be acid or base catalysts. The alkaline catalysts show high performance for obtaining vegetable oils with high quality, but a question often arises; that is, the oils contain significant amounts of FFA which cannot be converted into biodiesel but rather give a lot of soap. An alternative way of processing these vegetable oils is to use a Brønsted acid catalyst. Currently, the cataly sts more used in biodiesel production are the organic acids, such as the derivates of toluenesulfonic acid and, more often, mineral acids such as H_2SO_4. Sulfuric acid is one of the most common chemicals on earth. Concentrated sulfuric acid is used as a catalyst, and has a dual role which are speeds up the reaction and acts as a dehydrating agent, forcing the equilibrium to the right and resulting in a greater yield of ester. Only very small amounts of sulfuric acid are used, as large amounts would be wasteful, uneconomical, and would complicate the final separation processes.

2.11 Variables affecting the biodiesel production

Despite of catalyst, other variables affecting biodiesel production are temperature, catalyst loading, oil to methanol ratio, internal and external mass transfer resistance.

Study showed that optimum reaction conditions for free fatty acid reduction were investigated were found to be 65^{o}C, 15:1 methanol to oil ratio and 0.5 wt% sulphuric acid using fractional factorial design of experiment based on Taguchi method(Modhar A. Khan et al. 2010). In the absence of external mass transfer resistance, the rate of reaction is directly proportional to catalyst loading based on the entire liquid phase volume. The catalyst loading was varied over a range of 0.01–0.08 g/cm^3 on the basis of the total volume of the reaction mixture and the results are presented in figure below. The conversion of fatty acid increased with an increase in catalyst amount due to the proportional increase in the number of active sites. Further reactions were carried out with 0.041 g/cm^3 catalyst loading in all other experiments (Srilatha et al., 2012).

The effect of temperature on the esterification of FFA in rice bran was studied and the result show the increasing in the temperature accelerates the reaction. The catalyst also exhibited considerable conversion even at low reaction temperature suggesting the high activity of the catalysts (Srilatha et al., 2012).

The esterification of the FFAs present in WCO with methanol was catalyzed by 25 wt.% TPA/Nb_2O_5 catalyst at a methanol to WCO molar ratio of 14:1, reaction temperature of 65 °C with 1.65 wt.% (with respect to the initial oil mass) catalyst loading and from the results it could be observed that the fatty acid conversion increased gradually with time and remained almost constant beyond 5 h of reaction time (Srilatha et al., 2012) . From both studies, we can summarized that different catalyst have their own specific alcohol to oil ratio with particular temperature to achieve optimum yield of FAME.

3 MATERIALS AND METHODS

3.1 Overview

This paper presents the method to evaluate the performances of the reaction with three different parameters in similar condition by varying the value of each parameter to obtain the experimental data.

3.2 Introduction

This paper presents a a review of the esterification reaction, setup process and how the experiment is run in complete step by step procedures.

3.3 Chemicals

Chemicals used in this study is hydrochloric acid, HCl, sodium hydroxide, NaOH and sodium carbonate, Na_2CO_3. Concentration of HCl, NaOH and Na_2CO_3 used is 1.0 N. To prepare 1.0 N of NaOH solution, 4.0 grams of NaOH was dissolved in 100 mL of distilled water. For 1.0 N of Na_2CO_3 solution, 10.6 g of Na_2CO_3 was dissolved in 100 mL of distilled water.

3.4 Sample Preparation

The simulated cooking oil is prepared by adding oleic acid into the fresh vegetable oil. The amount of free fatty acids then can be controlled by the amount of added oleic acid.

3.5 Apparatus Setup

The equipment is set up in the fume hood for safety purpose as this experiments used some dangerous chemicals. The retort stand is to place the reactor and heating mantle also with the mechanical stirrer. The reactor is placed in heating mantle with it connected with condenser, also thermometer is placed at one of the hole with a stopper and the reactor head cover the reactor with a metal holder. The stirrer at the position which it accommodate with the stopper that has small hole at the centre. The apparatus set is shown in fig.3.5.

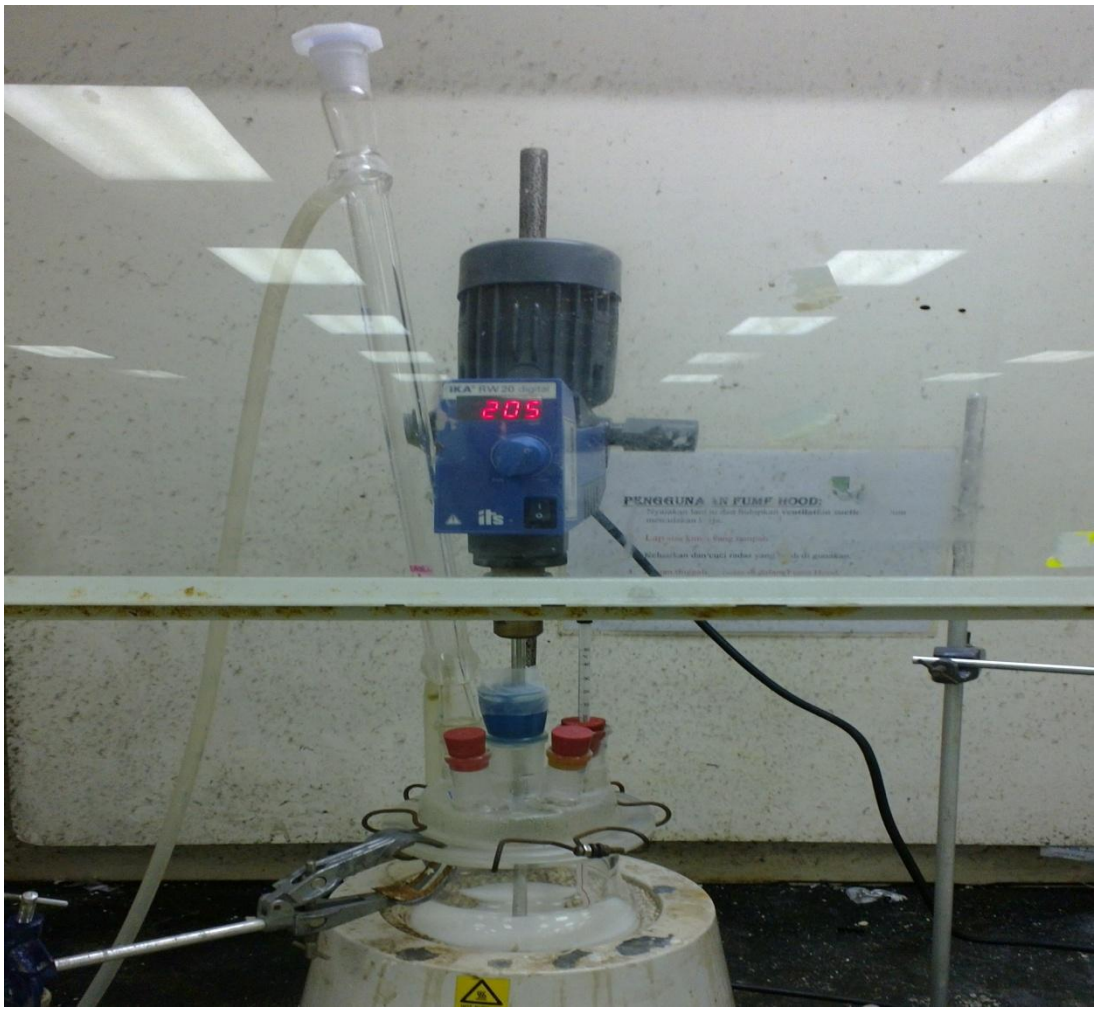

Fig3.5: Apparatus setup

3.6 Experiment procedure(esterification reaction)

First, Pre-heat the oil to remove water content. Then, Mix with methanol (different ratio) and stir (various rpm) in jacketed batch reactor at temperature= X (different temperature) add catalyst (vary the amount and size of particles). After that, take sample every half an hour for 3 hours. Directly after the sample is taken, the sample directly taken for analysis. At the end of reaction, sample left to settle down and two-layers of mixture is formed consist of pre-treated oil and methanol. Lastly, Oil rich region is separated using rotary evaporator and ready for transesterification.

3.7 Sample analysis and acid number determination

2g of sample is weight into a 250 ml Erlenmeyer flask. Next, the oil is now diluted with a titration solvent (100:99:1 mixture of Toluene, Isopropyl alcohol and water). Then, add 0.5 ml of indicator solution to the titration flask from a dropping bottle with a

graduated dropper. Swirl the solution to mix in the indicator, avoiding agitation. Finally, Load burette with 0.1MKOH in isopropanol and titration process ready to initiate with a bright orange starting color and a dark green/brown endpoint as shown in fig3.7. The Free Fatty Acid reading is taken.

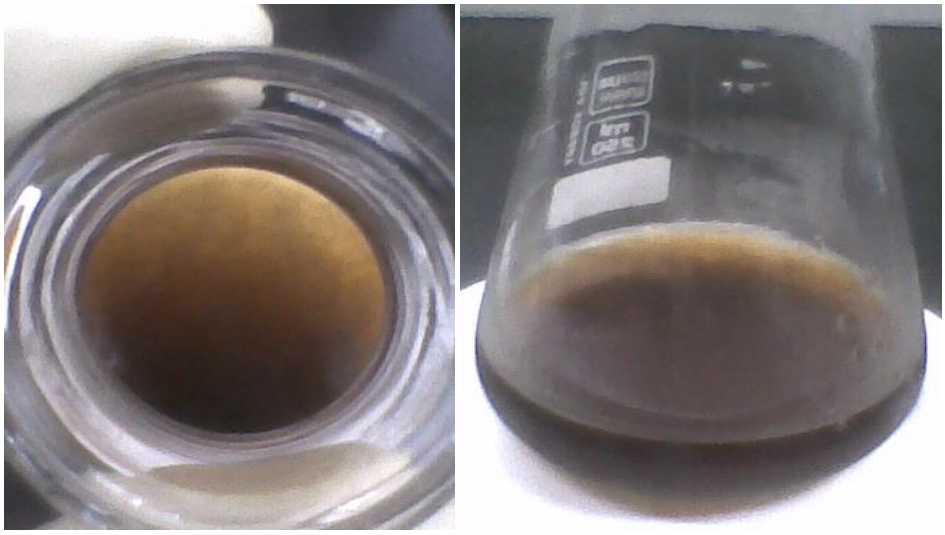

Fig3.7:Endpoint colour of titration

3.8 Free Fatty Acids Calculation

For the calculation, a blank titration is performed on 100 mL of the titration solvent and 0.5 mL of the indicator solution, adding 0.1-mL or less increments of the 0.1 M KOH solution. The titration solvent usually contains weak acid impurities which react with the strongly basic components of the sample. To correct the base number for the sample, an acid number blank upon the solvent is determined.

Calculate the acid number as follows:

Acid number, mg of KOH/g $= [(A - B)M \times 56.1]/W$ equation (1)

A = KOH solution required for titration of the sample, mL,
B = KOH solution required for titration of the blank, mL,
M = molarity of the KOH solution, and
W = sample used, g.

Free Fatty acid, % $= \frac{\text{Acid number ,mg of KOH /g}}{2}$ equation (2)

4 RESULTS AND DISCUSSION

4.1 Overview

This paper presents the results obtained from each experiment where with different parameters and each parameters the experiment conducted four times to observe and obtain the optimum condition. Three graphs represent the results.

4.2 Data on table(results)

Time (min)	FFA conversion(%)
0	0
30	75
60	96
90	96.5
120	97
150	0
180	75

Table 4.2a: Time Effect on Reaction

Free Fatty Acid Conversion (%)				
	Catalyst loading (wt%)			
Time (min)	0.3	0.4	0.5	0.7
0	0	0	0	0
30	30	39	50	50
60	44	57	69	70
90	55	70	72	72
120	65	72	80	81
150	70	75	85	86
180	73	80	90	90

Table 4.2b: Effect of catalyst loading

Free Fatty Acid Conversion (%)				
Time (min)	Methanol to oil molar ratio			
	5:01	10:01	15:01	20:01
0	0	0	0	0
30	30	39	50	50
60	44	57	69	70
90	55	70	72	72
120	65	72	80	81
150	70	75	85	86
180	73	80	90	90

Table 4.2c: Effect of methanol to oil molar ratio

4.3 Discussion

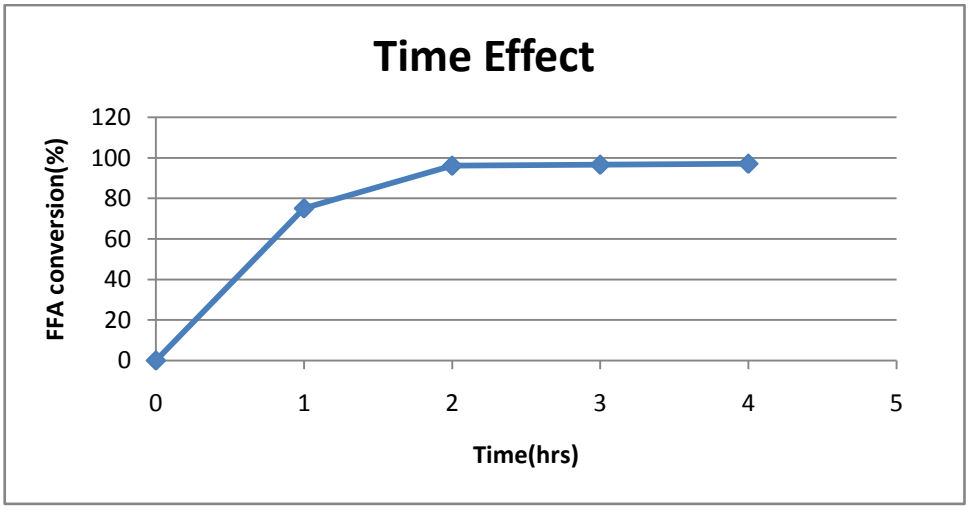

Fig.4.3a: Effect of time on conversion of FFA

The condition to study the effect of time on FFA conversion was set to be; temperature at 60^0C, 15:1 methanol to oil ratio and catalyst loading at 0.5wt%. The graph shows that the FFA conversion occurred at first two hours and then the line constant at conversion of 98%. The conditions were at optimum condition and thus the optimum conversion was at 98% conversion of FFA.